

[Second Edition.]



PATENT SPECIFICATION

Application Date : April 16, 1942. No. 5018/42.

560,529

Complete Specification Left : Dec. 23, 1942.

Complete Specification Accepted : April 7, 1944.

PROVISIONAL SPECIFICATION

Preparation of Stabilized Edible Substances.

I, HAROLD EDWIN POTTS, Chartered Patent Agent, of 12, Church Street, Liverpool, in the County of Lancaster, Subject of the King of Great Britain, do hereby declare the nature of this invention which has been communicated to me by Distillation Products Inc., a Corporation organised under the laws of the State of Delaware, United States of America, of 100, West Tenth Street, Wilmington, State of Delaware, United States of America, to be as follows :—

This invention relates to the preparation of stabilized edible substances and particularly stabilized edible substances in refined or deodorized condition.

It is known to incorporate various antioxidants in edible substances but most antioxidants heretofore used have been of questionable value because their effect upon the animal organism was not known. In many cases it appeared that many of these substances were poisonous or had a harmful effect. Other antioxidants have been exceedingly expensive.

According to the present invention, the edible substance to be stabilized is incorporated with scum derived from the vacuum-carrier gas treatment of a vegetable or animal oil which contains tocopherol and then the edible substance containing the scum is subjected to deodorization treatment, usually under vacuum.

The scum which is utilized in the invention is a substance which has heretofore been a waste by-product of the vegetable and animal oil (i.e. glyceride or fat) industry and particularly the vegetable and animal oil deodorization industry. This material is a complex mixture of organic compounds volatilized from the oil during deodorization and like procedures. Examples of such materials are those known in the deodorization art as "deodorizer hotwell sludge," "catch basin scum," "catch-all sludge," or "trap sludge." Application 2310/41 (Serial No. 548,731) contains examples of suitable sludges. It is to be understood that the word scum as used herein shall include all such matters in crude or partially refined condition produced by the

methods mentioned or by an equivalent method involving the separation of the organic material volatilized during the carrier gas, vacuum treatment of vegetable and animal oils which contain tocopherol.

Useful scum can only be obtained from the foregoing procedures when applied to oils which contain tocopherol. Most of the vegetable and animal oils in their fresh and natural condition contain this material, but the vegetable oils are usually considerably richer sources and it is preferred to utilize scum produced from them. Examples of particularly rich oils are corn, cotton-seed and soybean. Fresh oils are preferred as a source of the scum and particularly oils which have been subjected to no treatment which would have a harmful effect upon the tocopherol. Ordinarily alkali refining decreases the tocopherol content, but oils which have been alkali refined can serve as a useful source although a poorer one than the crude oils.

The scum as obtained usually contains about 5% to 20% water. Most of the water is preferably removed before use, though the invention is not restricted to this expedient. The water can be removed in a simple way by merely heating the scum which causes it to separate into an oil and water layer. The oil layer can then be withdrawn or decanted and is in substantially anhydrous condition.

The scum has among other things a high content of free fatty acids and organic quinones and is very corrosive. It is thus in the very nature of things contaminated with metals, particularly iron. In many cases it is desirable to remove the metals and their soaps by a preliminary treatment with a mineral acid such as sulphuric or hydrochloric. Also reduction such as with acid and zinc dust increases the tocopherol content by converting tocopherol oxidation products back into tocopherol.

The scum in wet or dried condition is then dissolved in or thoroughly intermingled with the edible material. When the invention is applied to the preparation of stabilized fats and oils, the scum is

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merely dissolved in the oil in considerable amounts. Examples of oils which can be stabilized are hydrogenated oil and fats such as hydrogenated cottonseed oil, soybean, corn, olive, etc. oils. The invention is of particular value in connection with the stabilization of oils which have been subjected to a treatment which would result in a large loss of the natural anti-oxidants frequently contained therein. Most refining operations destroy the natural antioxidants and the invention is of particular use in stabilizing such oils and fats. When the invention is applied to the stabilization of finely ground edible substances such as wheat germ, wheat middlings, ground corn germ, etc., it is desirable that the scum be substantially evenly distributed throughout the material. This can be brought about by stirring and thorough agitation during the mixing operation. Slight heating would have no undesirable effect and would increase the rate of distribution. It is desirable, but not essential, that the scum be treated to remove water as described above.

The scum usually contains about 1% to 6% tocopherol and should be incorporated in the substance to be stabilized in amounts sufficient to give the desired stability calculated on the tocopherol content. It will be realized that the scum contains the odoriferous materials contained in the oil from which it is derived and that consequently the higher the amount of scum incorporated the more complete must be the subsequent deodorization treatment, for example with carrier gas under vacuum. The amount of scum incorporated obviously depends upon the potency or tocopherol content of the scum, the character of the material to be stabilized, and its content of natural anti-oxidants. Up to about 35% of scum may be incorporated but much smaller amounts such as .001 to 10% are usually satisfactory depending of course upon the tocopherol content of the scum and the stability desired.

The mixture is then subjected to deodorization, preferably with a carrier gas under reduced pressure. This type of deodorization treatment is well known in the deodorizing art and need not be described in detail here. It is advisable to perform the deodorization under conditions insufficient to volatilize a substantial amount of the tocopherol. The deodorization usually involves heating the material to a temperature of about 200° C. and passing steam therethrough which has a temperature the same as or higher than that of the heated oil. The procedure is carried out under vacuum and in

the more recently improved deodorization processes the pressure is usually about 6 to 12 mm. The treatment requires several hours with batch processes but recent continuous methods result in deodorization in a shorter period of time. It is also satisfactory to deodorize the mixture under vacuum without a carrier gas as described in U.S. Patent No. 2,249,524 Hickman.

In connection with the solid substances which it is desired to stabilize, inert gas such as steam is passed therethrough in a heated or unheated container under reduced pressure. Steaming for ten minutes to three hours is usually sufficient.

The vacuum deodorization treatment has little or no destructive effect on the tocopherol incorporated in the mixture. However, the tocopherol is vaporizable under high vacuum conditions and the deodorization treatment should preferably be applied so that as little removal of tocopherol as possible takes place. Odoriferous materials have a substantially higher vapour pressure so that they can be preferentially removed.

While steam-vacuum deodorization constitutes the preferred embodiment of the invention, it is to be understood that other carrier gases such as hydrogen or carbon dioxide may be employed. Likewise, as mentioned, the deodorization may be accomplished by heating under relatively high vacuum without a carrier gas.

EXAMPLE 1.

Crude scum was warmed to form an aqueous and an oil layer. Two parts of the oil or scum containing 5% tocopherol were thoroughly incorporated with eighty parts of wheat middlings to yield a relatively palatable flaky mixture containing about 1% tocopherol. The mixture was then placed in a steam deodorizer and steam introduced or drawn continuously thereinto with stirring for two hours at a pressure of 55 mm. The middlings were withdrawn and were found to contain approximately 1% tocopherol and to have a good odour, and a free fatty acid content of less than 1%.

EXAMPLE 2.

20 parts of crude scum well heated to 50° C. and 100 parts of 10% HCl and 1 part of zinc dust were added with stirring. The mixture was settled for one hour and the acid layer removed. The upper layer was partly emulsified and was heated on a steam bath until the water layer separated. The oil layer was decanted washed and filtered. It contained 4.8% tocopherol and had a peroxide value of 4.6 milliequivalents (see "Rancidity in Vegetable Fats," Lea (1938), p. 107).

This scum was then added to a sample of prime steam lard to form a mixture containing .285% scum and containing .019% tocopherol. The mixture had a bacon like odour and taste not present in the original lard. The mixture was then steam deodorized at a temperature of 220° C., at 5 mm. pressure. The final product was free of odour and taste and had good stability as shown by the following data: 10

	Sample	% Tocopherol added in form of scum	"Swift Test" (See J. Oil & Soap 10 105 (1933))
15	Prime steam lard	None	3 hours
" "	"	.019	4 hours
" "	(steam deodorized)	.019	4 hours

The deodorized mixture contained 95% of the added tocopherol.

EXAMPLE 3.

To prime steam lard was added .745% of scum prepared as described in example 2 to give a mixture containing .03% tocopherol. The mixture was then treated with 2% of 20 Bé sodium hydroxide at a temperature of not over 60° C. The soaps were settled out and the liquid lard mixture clarified

by treatment with a neutral bleaching earth. The mixture was then steam deodorized as described in example 2. After two hours of steaming the product was odourless and tasteless. After four hours of steaming the odour and taste were the same as at 2 hours and only 5% of the added tocopherol had been removed. The stability was improved as shown by the following data.

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	Sample	% Tocopherol added in form of scum	"Swift Test"
40	Prime steam lard	None	8 hours
" "	"	.03	14 hours
45	" " (2 hours of steam deodorizing)	"	20 hours
" "	(4 hours of steam deodorizing)	"	20 hours

Dated this 15th day of April, 1942.

W. P. THOMPSON & CO.,
12, Church Street, Liverpool, 1,
Chartered Patent Agents.

COMPLETE SPECIFICATION

Preparation of Stabilized Edible Substances

I, HAROLD EDWIN POTTS, Chartered Patent Agent, of 12, Church Street, Liverpool, in the County of Lancaster, Subject of the King of Great Britain, do hereby declare the nature of this invention which has been communicated to me by Distillation Products Inc., a Corporation organised under the laws of the State of Delaware, United States of America, of 100, West Tenth Street, Wilmington, State of Delaware, United States of America, and in what manner the same

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is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the preparation of stabilized edible substances and particularly stabilized edible substances in refined and deodorized condition.

It is known to incorporate various anti-oxidants in edible substances but most 10 antioxidants heretofore used have been of questionable value because their effect upon the animal organism was not known. In many cases it appeared that many of these substances were poisonous or had a 15 harmful effect. Other antioxidants have been exceedingly expensive.

In application No. 548,731 of prior date hereto, in connection with which a specification was laid open to inspection before the date of the present application, there is described and claimed an improved procedure for preparing and employing anti-oxidants and improved anti-oxidant products wherein the anti-oxidant is the scum obtained by the carrier gas treatment at elevated temperature of vegetable or animal oils containing tocopherol.

It has already been proposed to winterize crude cottonseed oil, to add to the winterized product a further small amount of crude cottonseed oil, and to deodorize by a process such as blowing with steam, with or without vacuum.

It has also been proposed to treat a fatty material containing a natural anti-oxidant with a solvent, to separate the extract containing the anti-oxidant, to add it to a substance of fatty nature 40 having an undesirable odour and to heat the mixture to 110°—135° C. to drive off the objectionable odours.

According to the present invention a process of preparing a food is provided 45 which comprises incorporating with an edible substance a crude or partially refined scum derived from the vacuum carrier gas treatment of crude vegetable or animal oils containing tocopherol, and subjecting the mixture to deodorization under conditions so mild as to remove the odoriferous constituents from said scum without substantially affecting the tocopherol.

The process is preferably applied to an 55 edible glyceride. The mixture of glyceride and scum can be refined by treating with alkali before or after deodorization. A rather dilute caustic alkali may be added 60 to the mixture in liquid condition, the whole stirred at a slightly elevated temperature (about 50° to 60° C.) and the soap stock thus produced allowed to settle. The supernatant refined oil is decanted 65 or siphoned off, exposure to the air being

avoided as much as possible during this treatment. The product of deodorization and alkali refining or alkali refining and deodorization may be added to other foods and serves as a source of vitamin E.

The invention also includes a process of preparing a food which comprises incorporating with an edible substance a crude or partially refined scum obtained from the vacuum carrier gas treatment of crude animal or vegetable oil containing tocopherol, and subjecting the mixture to reduced pressure and elevated temperature to remove the odoriferous constituents in vapour form without affecting the tocopherol.

In some cases the colour of the product may be dark or otherwise not exactly as desired and the mixture may be improved in colour by filtering it through an activated earth, activated carbon or equivalent decolorizing agent.

The scum which is utilized in the invention is a substance which has heretofore been a waste by-product of the vegetable and animal oil (i.e. glyceride or fat) industry and particularly the vegetable and animal oil deodorization industry. This material is a complex mixture of organic compounds volatilized from the oil during deodorization. Examples of such materials are those known in the deodorization art as "deodorizer hotwell sludge," "catch basin scum," "catch-all sludge," or "trap sludge." It is to be understood that the word scum as used herein shall include all such matters in crude or partially refined condition produced during the carrier gas, vacuum treatment of vegetable and animal oils which contain tocopherol.

Useful scum can only be obtained from the foregoing procedures when applied to oils which contain tocopherol. Most of the vegetable and animal oils in their 110 fresh and natural condition contain this material, but the vegetable oils are usually considerably richer sources and it is preferred to utilize scum produced from them. Examples of particularly rich oils 115 are corn, cotton-seed and soybean. Fresh oils are preferred as a source of the scum and particularly oils which have been subjected to no treatment which would have a harmful effect upon the tocopherol. Ordinarily alkali refining decreases the tocopherol content, but oils which have 120 been alkali refined can serve as a useful source, although a poorer one than the crude oils.

The scum as obtained usually contains about 5% to 20% water. Most of the water is preferably removed before use, though the invention is not restricted to this expedient. The water can be removed 130

in a simple way by merely heating the scum which causes it to separate into an oil and a water layer. The layer can then be withdrawn or decanted and is in substantially anhydrous condition.

The scum has among other things a high content of free fatty acids and organic quinones and is very corrosive. It is thus in the very nature of things contaminated with metals, particularly iron. In many cases it is desirable to remove the metals and their soaps by a preliminary treatment with a mineral acid such as sulphuric or hydrochloric. Also reduction such as with acid and zinc dust increases the tocopherol content by converting tocopherol oxidation products back into tocopherol.

The scum in wet or dried condition is then thoroughly intermingled with the edible substance to be stabilized. If this is an edible glyceride the scum can simply be dissolved in it. Examples of oils which can be stabilized are hydrogenated oils and fats such as hydrogenated cottonseed, soybean, corn or olive oil. The invention is of particular value in connection with the stabilization of oils which have been subjected to a treatment which would result in a large loss of the natural antioxidants frequently contained therein. Most refining operations destroy the natural antioxidants and the invention is of particular use in stabilizing such oils and fats. When the invention is applied to the stabilization of fine ground edible substances such as wheat germ, wheat middlings or ground corn germ, it is desirable that the scum be substantially evenly distributed throughout the material. This can be brought about by stirring and thorough agitation during the mixing operation. Slight heating would have no desirable effect and would increase the rate of distribution. It is desirable, but not essential, that the scum be treated to remove water as described above.

The scum usually contains about 1% to 6% tocopherol and should be incorporated in the substance to be stabilized in amounts sufficient to give the desired stability calculated on the tocopherol content. It will be realized that the scum contains the odoriferous material contained in the oil from which it is derived and that consequently the higher the amount of scum incorporated the more complete must be the subsequent deodorization treatment, for example with carrier gas under reduced pressure. The amount of scum incorporated obviously depends upon the potency or tocopherol content of the scum, the character of the material to be stabilized, and its content

of natural anti-oxidants. Up to about 35% of scum may be incorporated but much smaller amounts such as .001 to 10% are usually satisfactory depending of course upon the tocopherol content of the scum and the stability desired.

The mixture is then subjected to deodorization, preferably with a carrier gas under reduced pressure. This type of deodorization treatment is well known in the deodorizing art and need not be described in detail here. It is essential to perform the deodorization under conditions insufficient to volatilise a substantial amount of the tocopherol. The deodorization usually involves heating the material to a temperature of about 150—250° C. and passing steam therethrough which has a temperature the same as or higher than that of the heated oil. The procedure is carried out under reduced pressure and in the more recently improved deodorization processes the pressure is usually about 8 to 12 mm. The treatment requires several hours with batch processes but recent continuous methods result in deodorization in a shorter period of time. It is also satisfactory to deodorize the mixture under vacuum without a carrier gas by using short path distillation at a pressure below 0.1 mm. as described in U.S. Patent No. 2,249,524 Hickman; in that specification the short path distillation is for deodorizing vitamin containing oils but as it results in obtaining the odour and taste materials in a distillate containing only 0.5 and 2% of the vitamin contents of the oil leaving the purified oil as undistilled residue, the process is one which is applicable in carrying out the present invention.

The vacuum deodorization treatment has little or no destructive effect on the tocopherol incorporated in the mixture. However, the tocopherol is vaporizable under high vacuum conditions and the deodorization treatment is, therefore applied under conditions so mild that as little removal of tocopherol as possible takes place. Odoriferous materials have a substantially higher vapour pressure so that they can be preferentially removed.

EXAMPLE 1.

20 parts of crude scum were heated to 50° C. and 100 parts of 10% HCl and 1 part of zinc dust were added with stirring. The mixture was settled for one hour and the acid layer removed. The upper layer was partly emulsified and was heated on a steam bath until the water layer separated. The oil layer was decanted, washed and filtered. It contained 4.8% tocopherol and had a peroxide value of 4.6 milliequivalents (see 130

"Rancidity in Vegetable Fats," Lea (1938), p. 107).

This scum was then added to a sample of prime steam lard to form a mixture containing .285% scum and containing .019% tocopherol. The mixture had a

bacon like odour and taste not present in the original lard. The mixture was then steam deodorized at a temperature of 220° C., at 5 mm. pressure. The final product was free of odour and taste and had good stability as shown by the following data:

15	Sample	% Tocopherol added in form of scum	"Swift Test" (See J. Oil & Soap 10 105 (1933))
	Prime steam lard	None	3 hours
	" " "	.019	4 hours
	" " " (steam deodorized)	.019	4 hours ^a

20 The deodorized mixture contained 95% of the added tocopherol.

EXAMPLE 2.

To prime steam lard was added .745% of scum prepared as described in example 25 I to give a mixture containing 0.3% tocopherol. This mixture was then treated with 2% of 20 Bé sodium hydroxide at a temperature of not over 60° C. The soaps were settled out and 30 the liquid lard mixture clarified by treat-

ment with a neutral bleaching earth. The mixture was then steam deodorized as described in example 1. After two hours of steaming the product was odourless and tasteless. After four hours of steaming the odour and taste were the same as at 2 hours and only 5% of the added tocopherol had been removed. The stability was improved as shown by the following data:

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45	Sample	% Tocopherol added in form of scum	"Swift Test"
	Prime steam lard	None	8 hours
	" " "	.03	14 hours
	" " " (2 hours of steam deodorizing)	"	20 hours
	" " " (4 hours of steam deodorizing)	"	20 hours

50 EXAMPLE 3.

Eighty parts of butter fat are mixed with 20 parts of refinery scum containing approximately 3 per cent. of tocopherol. The mixture is placed in a steam deodorizer and deodorized at 5 mm. for 4 hours at 200° C. until the acid value is substantially lowered. The mixture is then cooled and transferred to a refining kettle where 2 parts of caustic soda, 20% solution, are stirred in. After settling, the clear supernatant fat is filtered through a mixture of 3 parts of activated clay and 1 part of activated carbon. The final product contains 1/2% of mixed toco-

pherols. It is suitable for adding to butter or margarine in the proportion of 1 part to 10—250 parts of butter.

EXAMPLE 4.

Eighty parts of scum containing 2% of mixed tocopherols are mixed with 20 parts of prime lard and the mixture is placed in a steam deodorizer and deodorized at about 5 mm. for four hours at 200° C. The product is then cooled, transferred to a refining kettle where two parts of caustic soda in the form of a 20% solution are stirred in. The soap stock is permitted to settle and the clear, supernatant fat is withdrawn and filtered as

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- described above. The finished product is then added to margarine to provide a product containing about .02% mixed tocopherol. Ten thousand units of vitamin A per pound of margarine were also incorporated. The tocopherol will substantially preserve the vitamin A and at the same time acts as a useful form of vitamin E in the human diet.
- I am aware that application No. 126/42 (Serial No. 557,872) claims a process of preparing a human or animal food which consists in incorporating with subdivided solid vegetable matter, a crude scum containing fatty acids obtained by the vacuum carrier gas treatment of crude animal or vegetable oils containing tocopherol, and driving off a part of the fatty acids by drawing steam under vacuum continuously therethrough and I make no claim therefor in the present application. Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, as communicated to me by my foreign correspondents, I declare that what I claim subject to the foregoing disclaimer is:—
1. Process of preparing a food which comprises incorporating with an edible substance a crude or partially refined scum derived from the vacuum carrier gas treatment of crude animal or vegetable oil containing tocopherol and subjecting the mixture to deodorization under conditions so mild as to remove the odoriferous constituents from the scum without substantially affecting the tocopherol.
 2. Process as claimed in claim 1 applied to edible glycerides.
 3. Process as claimed in claim 2 in which an alkali refining step is inserted before or after the deodorization step.
 4. Process as claimed in claim 2 or 3 in which the deodorized mixture is treated with a decolorization agent.
 5. Process as claimed in any of claims 1 to 4 in which steam under reduced pressure is used as deodorizing agent.
 6. Process as claimed in claim 5 in which the deodorization treatment occurs at a temperature of between 150° and 250° C. and at a pressure of less than 12 mm.
 7. Process as claimed in any of the preceding claims in which the scum is reduced before incorporation.
 8. Process as claimed in any of the preceding claims in which the scum is treated with acids before incorporation.
 9. Process as claimed in any of the preceding claims in which the scum is dehydrated before incorporation.
 10. Process as claimed in claim 1, which comprises incorporating with an edible substance a crude or partially refined scum obtained from the vacuum carrier gas treatment of crude animal or vegetable oil containing tocopherol and subjecting the mixture to reduced pressure and elevated temperature to remove the odoriferous constituents in vapour form from the scum without substantially affecting the tocopherol.
 11. An edible substance whenever treated by the methods of the preceding claims.

Dated this 22nd day of December, 1942.

W. P. THOMPSON & CO.,
12, Church Street, Liverpool, 1,
Chartered Patent Agents.

Abingdon : Printed for His Majesty's Stationery Office, by Burgess & Son.
(Wt. 8213A.—4/1948.)

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